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EXECUTIVE SUMMARY

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PERFORMANCE OF ALLOY 800 IN STEAM GENERATOR ENVIRONMENTS: A LITERATURE SURVEY

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The steam generators at Darlington NGS use an iron-nickel-chromium alloy, Incoloy 800, as the tubing material. This is the first time that Ontario Hydro utilizes this alloy in a nuclear station for this type of application. While there is ample field experience on the operating performance of alternative alloys such as Inconel 600, the information on alloy 800 is far more limited. This report briefly summarizes the available information on the operating experience on I800, and points out the areas in which further work is required.

There is no operating or laboratory evidence which suggests the premature corrosion failure of nuclear-grade I800 under normal CANDU steam generator operating parameters. However, in laboratory tests, sea water in-leakage has caused tubing and support structure damage in restricted regions where impurities concentrate. Acidified fresh water in-leakage also produced corrosion damage but of a more restricted nature.

KWU has used this alloy in recirculating steam generators. The operating experience spans over the last 13 years with only one tube failure. The type of water treatment used by KWU is mostly coordinated phosphates. The type of water treatment is particularly important in the chemistry of localized regions where concentration of non-volatile impurities may take place because of the local thermalhydraulic conditions. Water treatment with phosphate provides a buffering effect in these local chemistries. Water treatment with volatile amines (AVT) of t h e type used by Ontario Hydro does not. Operating experience of units with one type of water treatment may not be necessarily relevant to units with the alternate chemistry.

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There is limited information on the performance of the alloy under AVT operating chemistry in the event of fresh water in-leakage, particularly the anodic polarization effect that the presence of oxygen may have on the corrosion rate. Information is also limited on the specific impact of these conditions on the corrosion in restricted geometries in a CANDU design.

Another area which requires further work is the effects of chemical excursions on materials in confined regions. It is important to have information on these effects as well as on possible remedial actions to minimize the resulting material damage. The possible galvanic effect of stainless steel 410 on the corrosion of I800 in these severe environments should also be investigated.

In view of the evidence available, two research programs are being undertaken by Research Division with funding from COG (CANDU Owners Group):

1) The investigation of the effects in confined regions of fresh water and dissolved oxygen in-leakage on stressed and unstressed I800 under AVT water and CANDU steam generator operating conditions.

2) The investigation of the effects in confined regions of water treatment plant chemical excursions (alkaline and acidic) and of possible mitigating actions (use of hideout return and neutralizers).

The results of these programs should provide operating guidelines as well as actions to be considered in the event of some abnormal secondary side chemistries.

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PERFORMANCE OF ALLOY 800 IN STEAM GENERATOR ENVIRONMENTS: A LITERATURE SURVEY

F. Gonzalez

The reported performance of alloy 800 in laboratory and operating plant environments has been excellent to date. However, there is limited information on the effects of chemical excursions and fresh-water in-leakage on this tubing material under steam generator operating conditions. In view of the literature surveyed, two research programs are proposed to complement the available information. The results of these programs should provide operating guidelines as well as actions to be considered in the event of certain abnormal secondary side chemistries.

INTRODUCTION

A request was made by TTSD to carry out a literature survey on the corrosion of titanium-stabilized Incoloy 800 (referred to as I800 or alloy 800) in nuclear steam generator environments. The request came as a follow-up of a previous memorandum/1/. The objective of the survey was to determine the usefulness of laboratory testing of nuclear grade I800 in view of the available literature results.

A computer literature search of recent references on the high temperature corrosion behaviour of alloy 800 was carried out using two data banks: Metadex and Chemical Abstracts. The search, which was extended from 1972, generated more than 900 references. Ninety references were found to be directly relevant to the objective of the survey. Folders with the results of the search and the relevant references are available from the author.

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BACKGROUND

Alloy 800 is a most important alloy in high temperature engineering including petrochemical processing and particularly electrical power generation. The use of alloy 800, as of any high temperature material designed to be used for long periods of time (30 years or more), requires that the plant be designed in the light of the best possible forecast of the behaviour of these materials under the expected operating conditions of stress, temperature and environment. Since proven observation and measurement of 30-year properties of materials cannot be easily available, it is necessary to adopt methods of prediction based on extrapolation of shorter term observations. An additional difficulty arises when trying to evaluate the performance of the material in prototype plants with changing environments which are not well defined.

In 1949 INCO introduced alloy 800 in the U.S.A. at a time when there was a nickel shortage due to the repercussions of the war economy. Alloy 800 was an inexpensive replacement for Inconel 600 (I600) for the sheathed elements of electric appliances. Users soon realized the remarkable properties of the alloy, its excellent oxidation resistance and fabrication characteristics. The incorporation of titanium and aluminum, which was a fortuitous consequence of the use of superalloy scrap in the manufacture, revealed an improvement in the material properties. The concentration of these and other minor alloying elements was later refined and alloy 800 has come to embrace a range of rather different materials/2/.

CORROSION RESISTANCE TO AGGRESSIVE ENVIRONMENTS

Initial studies on the corrosion of I800 were often reported without specification of the microstructure and metallurgical state of the sample alloys tested. This produced additional difficulties in the data interpretation and, in some cases, led to some conclusions on the material performance which were later negated by the operating experience. This situation has not been unique to I800; as an example, the pure-water high-temperature cracking of I600, the most commonly used alloy in PWRs, is presently a serious concern in the performance of nuclear steam generators. Although the phenomenon was known, it was not considered of sufficient importance to preclude the use of I600 as tubing material/3/.

The characterization of I800 corrosion initially dealt with its behaviour in aggressive environments. Its manufacturer, Huntington (INCO) alloys /4/, reported the following properties for the alloy: very resistant to atmospheric corrosion even in marine environments, resistant to sulphuric acid but only at low temperatures, vulnerable to hydrochloric and hydrofluoric acids

but fairly resistant to nitric acid. The risk of sensitization if heated at 540 to 760 °C is mentioned. Sensitization made the alloy susceptible to intergranular attack. The alloy was subject to pitting in strong neutral and acid chloride solutions. All these data were obtained at temperatures less than 100 °C.

The nuclear industry required standards for detecting the susceptibility to intergranular attack of nickel-based alloys. The development of these standards provided the means of comparatively rating the performance of the alloys available/5/.

Early surveys on the performance of alloy 800 with respect to stress corrosion cracking/3,6,7,8/ reported quite a satisfactory resistance of this alloy to cracking in deaerated caustic solutions; however, I600 outperformed it under the same conditions. In aerated caustic solutions, I800 was less susceptible to cracking than I600. There was evidence of chloride stress corrosion cracking in steam generator environments in the heat affected zone of some I800 welds. This indicated a reduced resistance to cracking in the sensitized material/9/.

The relative resistance of alloy 800 to stress corrosion cracking, mostly inferred from laboratory test results, and the optimized performance of the fine-grained, fully titanium-stabilized grade made the alloy attractive for the use in nuclear steam generators. Siemens originally used the alloy /10/. AECL considered the change from I600 to I800 to reduce radiation fields/11/. Previously, I600 had been used instead of Monel 400 mainly because its much lower corrosion rate, particularly at the higher concentrations of dissolved oxygen expected in the primary coolant in CANDU reactors with closed cycle boiling operation/12,13/.

EFFECT OF SPECIFIC IONS ON I800 PITTING

Reference 14 contains an extensive study of the pitting of I800 in several environments. The main conclusions of the work are summarized below. The variation of the pitting potential with several variables shows that there is a temperature (the "reversing temperature") at which the I800 pitting potential reaches a minimum value and pitting takes place at a maximum rate.

Pit morphologies also change with temperature in the range between 25 and 200 °C. At low temperatures, pits are localized and clean. The unaffected areas appear shiny. At higher temperatures the pit density increases and the pits are filled with chromium-rich corrosion products; the overall surface of the sample appears darker. In all pits there is an apparent increase of chromium at the bottom of the pit even at low temperatures.

The morphology of the pits encountered in the field corresponds to that at the intermediate temperatures of the range investigated.

The effect of chloride is interesting. Even high concentrations (3550 mg/kg) do not produce pitting of I800 at high temperatures when the electrochemical potential is kept sufficiently low. However, 20 mg/kg chloride concentrations can produce pits in the presence of copper ions. The presence of oxidizing species such as $CuCl_2$ or perhaps Fe_2O_3 changes the corrosion potential and induces pitting even at low chloride concentrations.

Under alkaline conditions (pH >10) I600 offers the best resistance to pitting. At lower pH, I800 shows better resistance than I600. The effects of sulphate, bicarbonate and phosphate are complex. These species inhibit the initiation of chloride-induced pitting but repassivation of existing pits becomes much more difficult with sulphate at steam generator layup or shutdown conditions; the presence of molybdates in solution apparently has the same inhibiting effect with respect to pitting as the presence of molybdenum in the alloy. Pit repassivation is accomplished more rapidly at temperatures greater than 200 ^{o}C.

Neutron irradiation affects some material properties of I800. For example, the fracture toughness response, which is excellent for this alloy, is significantly reduced after irradiating. However, this is not a concern for steam generator use /15/.

I800 IN NUCLEAR STEAM GENERATORS

PWR steam generators have experienced numerous failures because of the corrosion of the alloy tubing. The attack has mostly originated on the secondary side/16/. Causes of the attack are related to the in-leakage of condenser cooling water and dissolved oxygen as well as breakdown products from resins of the water treatment plant. Copper ions can enter the feedwater because of corrosion of copper bearing alloys in the feedtrain. These and other oxidizing species, for example ferric ions, have also detrimental effects since they change the electrochemical potential of the alloy to values where the corrosivity of the environment becomes unacceptable.

An interesting version of alloy 800 to the power industry is alloy 800H which is a grain size and carbon variant with improved creep rupture properties. It has found applications in high temperature gas (helium) cooled nuclear power plants in superheater and reheater tubes as a substitute for type 304 stainless steel/2/. Another version, presently known as "nuclear grade", presents an enhanced resistance to stress corrosion cracking which is achieved by a low carbon, fully-stabilized structure. This alloy has been selected as the steam generator

tube material for the Superphenix sodium-cooled fast-breeder reactor/2/.

NUCLEAR GRADE ALLOY 800

The nuclear-grade alloy 800 has a narrower composition range than that for alloy 800 as given by ASTM B 163-66. CANDU units use the AECL specification for Ni-Fe-Cr steam generator tubing also known by its Sandvik trademark name of Sanicro 30 /17/. The specifications of Sanicro 30, shown in table 1, are those of the nuclear grade material. KWU has used this alloy in recirculating steam generators as well. The alloy has given a satisfactory performance to date/16/.

A significant number of the research studies on the performance of alloy 800 have used the non-nuclear grade which has worse corrosion performance than its nuclear counterpart/18/. There is no conclusive evidence that demonstrates that nuclear-grade alloy 800 undergoes intergranular cracking under stress in high temperature pure water, in fact only sensitized material has failed in high purity oxygenated water environments. Non-sensitized alloy 600 on the other hand does crack in demineralized de-oxygenated water at 350 ^{o}C (Coriou effect).

TABLE 1

NUCLEAR GRADE INCOLOY 800 COMPOSITION

Ni	Cr	Fe	C	Mn	S	Si	Cu
32-35	20-23	Rem.	0.03	0.4-1.0	0.015	0.3-0.7	0.75max

Ti	Al	P	Co	N
0.60	0.15-0.45	0.20max	0.10max	0.03max

In steam, alloy 800 is quickly covered with a strongly adherent oxide layer; however, chromium and manganese depletion on the surface causes an acceleration of the rate of corrosion and even gives rise to intergranular attack. Circumstances which may encourage the diffusion of these two elements to the surface increase the resistance of alloy 800 to corrosion. A fine grained structure is therefore more resistant to corrosion since it has a large number of grain boundaries along which chromium and manganese can diffuse.

Standard alloy 800 is more susceptible to sensitization than the nuclear grade because of the restricted carbon content and higher stabilization ratio (Ti/C >12) of the latter. There is

experimental evidence that indicates that the resistance to stress corrosion cracking in high-chloride aerated solutions at high temperature is also improved in the nuclear grade type /19/.

When compared to Inconel 690 (I690), nuclear grade I800 performs quite similarly. Neither alloy is fully resistant to stress corrosion cracking in greater than 10% caustic deaerated solutions; however, alloy 690 appears to be less influenced by the stress levels. Similarly, alloy 690 is considered to be immune to high-temperature chloride-induced stress corrosion cracking; nuclear grade alloy 800 is not immune but it has a remarkably high resistance.

Based on the comparative performance in laboratory tests, nuclear grade I800 is considered to have an overall corrosion resistance comparable to that of alloy 690 which is presently the favoured choice for steam generator tubing/20/. In addition to the supposedly lower resistance to chloride cracking of nuclear grade I800, concerns have been raised about its higher coefficient of expansion (about 10% higher) when compared to that for I690 (I690 expansion coefficient is very similar to that of carbon steel). This has been given as an additional reason to prefer I690 over I800 /20/. The area in the operating unit which may be most affected is the expanded region near the top of the tubesheet. At the unit operating temperature the tube/tubesheet crevice is likely to be closed. At lower temperatures the crevice will tend to open. This may allow secondary water into the crevice.

Carbon steel, which has been used in I600-tubed nuclear steam generators, offers a certain degree of galvanic protection to the alloy tube in creviced regions. The change to a more corrosion resistant alloy such as 410 stainless steel (SS410) used in the support structures at Darlington, may actually change the galvanic coupling. There is some evidence in the literature that, in aggressive environments, pitting of I600 can be induced by the galvanic effect of SS410/21/. A similar effect could be possible on I800.

OPERATING EXPERIENCE OF ALLOY 800

The operating experience of KWU-built steam generators spans over 14 years with only one tube failure. This failure was diagnosed as secondary side intergranular attack in the sludge pile region /16,18/. The type of secondary water treatment is mostly coordinated phosphates. There is evidence of tube wastage resulting from the type of water treatment; however, a preventive sludge cleaning and tube plugging program has minimized its impact on the operating record /20,22,23/. There has been no indication so far of chloride-induced cracking or pure-water cracking.

The type of water treatment is particularly important in the chemistry of localized regions where concentration of non-volatile impurities may take place because of the local thermalhydraulic conditions. Water treatment with phosphate has a buffering effect in these local chemistries. Water treatment with volatile amines (AVT) of the type presently used by Ontario Hydro does not. Operating experience of units with one type of water treatment may not be necessarily relevant to units with the alternate chemistry. Only one unit, KWO Obrigheim, tubed with I800, utilizes AVT in its feedwater chemistry. Its reported operating experience has been excellent but quite limited/16/.

LABORATORY SIMULATION OF OPERATING PERFORMANCE

Laboratory investigations have been carried out to characterize the performance of alloy 800 under simulated operating conditions and abnormal chemistries which may occur in an operating unit. The emphasis of the investigation was mostly placed on the simulation of sea-water cooled stations.

Experimental results show that the effect of deaerated sea-water in-leakage on alloy 800 in heated crevices under CANDU operating conditions produced only micropitting (less than 50 μm); however, the crevice environment was quite aggressive to carbon steel inserts/24/. Residual phosphate treatment showed a strong mitigating effect on the crevice corrosion; however, AVT treatment did not prevent significant corrosion rates /25/. The effect of deaerated fresh-water was similar to that of sea-water on the tube alloy but less aggressive on the carbon steel. The pits on the tube alloy did not grow with time in either case /24/.

Model boiler tests with de-oxygenated acidified fresh-water also produced shallow pitting on I800 but no cracking/26/. The performance of highly stressed alloy 800 in oxygenated (1 ppm oxygen) AVT water contaminated with 10 ppm sodium hydroxide was excellent at high temperature. No stress corrosion cracking could be detected after a period of one and a half years/27/.

Keeping the dissolved oxygen concentration low in the steam generator water is extremely important since its effect is similar to that of anodic polarization. The simultaneous presence of dissolved oxygen and chlorides can make I800 quite vulnerable to other types of corrosion such as pitting attack despite its intrinsic high resistance to pitting/28/.

Other oxidants such as copper (II) chloride have been shown to be more aggressive to I800 than oxygen in high temperature water but this chemical species is unlikely to be present in all-ferrous feedtrain systems/29/.

CONCLUSIONS AND RECOMMENDATIONS

Titanium-stabilized I800 is an excellent alloy for use in nuclear steam generators. There is no operating or laboratory evidence which suggests corrosion failure under normal CANDU steam generator operating parameters. However, sea-water in-leakage may cause tubing and support structure damage in restricted regions where impurities concentrate. Acidified fresh-water in-leakage may also produce corrosion damage but of a more restricted nature.

There is limited information on the performance of the alloy under AVT operating chemistry in the event of fresh-water in-leakage, particularly the anodic polarization effect that the presence of oxygen may have on the corrosion rate. Information is also limited on the specific impact of these conditions on the corrosion in restricted geometries in a CANDU design. Laboratory results on the effect of dissolved oxygen on the corrosion performance of I600 have shown that this effect may be quite important on localized corrosion particularly under sludge piles/30/.

There is, therefore, a need to investigate this effect in suitably designed corrosion probes and vessels that duplicate the design and operating parameters of CANDU steam generators. The chemistry in these localized regions can thus be duplicated as well. A similar recommendation on the effect of oxygen and condenser in-leakage on cracking resistance has been made elsewhere/31/.

The difference between the expansion coefficient of I800 and carbon steel is of particular concern. There is need to find out the effects of temperature cycling on the potential for concentrating impurities of the expanded tube sections in the tubesheet and concomitant corrosion effects.

Another area which requires further work is the effects of chemical excursions on materials in confined regions. Sulphuric acid may cause pitting, general attack and possibly intergranular attack on I800 tubing. It may also induce cracking. However, the concentration limits that are unacceptable are not well determined. Since these excursions are known to occur in feedtrain systems, it is important to have information on these effects as well as on possible remedial actions to minimize the resulting material damage. The possible galvanic effect of SS410 on the corrosion of I800 in these severe environments should also be ascertained.

In view of the evidence available, two research programs are proposed:

(1) The investigation of the effects in confined regions of fresh-water and dissolved oxygen in-leakage on stressed and unstressed I800 under AVT water and CANDU steam generator operating conditions.

(2) The investigation of the effects in confined regions of water treatment plant chemical excursions (alkaline and acidic) and of possible mitigating actions (use of hideout return and neutralizers).

The results of these programs should provide operating guidelines as well as actions to be considered in the event of some abnormal secondary side chemistries.

These programs will be undertaken by Research Division with COG funding (programs 1904 and 1905).

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